IS 5062-3 (1969): Methods of test for brown coals and lignites, Part III: Determination of the yields of tar, water, gas and coke by low temperature distillation [PCD 7: Solid Mineral Fuels]

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IS : 5062 (Part III) - 1969
(Reaffirmed 2000)

Indian Standard

METHODS OF TEST FOR BROWN COALS AND LIGNITES

PART III DETERMINATION OF THE YIELDS OF TAR, WATER, GAS AND COKE BY LOW TEMPERATURE DISTILLATION

(First Reprint NOVEMBER 1984)

UDC 662.642:662.715

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *June* 1969

Indian Standard

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METHODS OF TEST FOR BROWN COALS AND LIGNITES

PART III DETERMINATION OF THE YIELDS OF TAR, WATER, GAS AND COKE BY LOW TEMPERATURE DISTILLATION

0. FOREWORD

0.1 This Indian Standard (Part III) was adopted by the Indian Standards Institution on 25 March 1969, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 The yields of the distillation products by low temperature distillation, especially the yield of tar, forms the basis for the classification of brown coal and lignite for use in low temperature carbonization. It has been experimentally seen that for evaluating brown coals or lignites for the manufacture of briquetted smokeless fuel the modified Fischer method described in this standard yields truer values than the conventional method.

0.3 This method is based on the corresponding Draft recommendation No. 865 prepared by TC/27 ' Soild Mineral Fuels ' of the International Organization for Standardization (ISO).

0.4 In reporting the results of an analysis made in accordance with this standard, if the final value observed or calculated is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes the method for the determination of the yields of tar, water, gas and coke obtained from brown coal and lignite by distillation to a final temperature of 520°C.

2. PRINCIPLE

2.1 The sample is heated in an aluminium retort to a temperature of 520°C for a period of 80 minutes. The products of decomposition pass

*Rules for rounding off numerical values (*revised*).

into a water cooled receiver; the tar and water are condensed while gaseous products escape to atmosphere. The coke residue remaining in the retort is weighed. The receiver and its contents are also weighed and the weight of the water in it determined by distillation; the weight of tar is obtained by difference. The total water in the receiver includes the moisture in the coal as well as that from the decomposition of the coal and a separate determination of moisture in the coal, by distillation, is made so that the decomposition water can be calculated.

2.2 The amount of gas (also including errors of determination) is obtained by subtracting the sum of the percentages of coke, tar and decomposition water from 100. The results are reported on the 'as analysed' and on the 'dry' basis.

3. APPARATUS

3.1 Retort — An aluminium retort with the dimensions shown in Fig. 1, with the cover fitted, its capacity without the outlet tube is 170 ± 10 ml; the outlet tube is made of brass and its internal wall is clean and polished.

3.2 Heating Furnace — A furnace heated either electrically or by gas. For electrical heating, a resistance wire furnace or a silicon carbide rod furnace may be used.

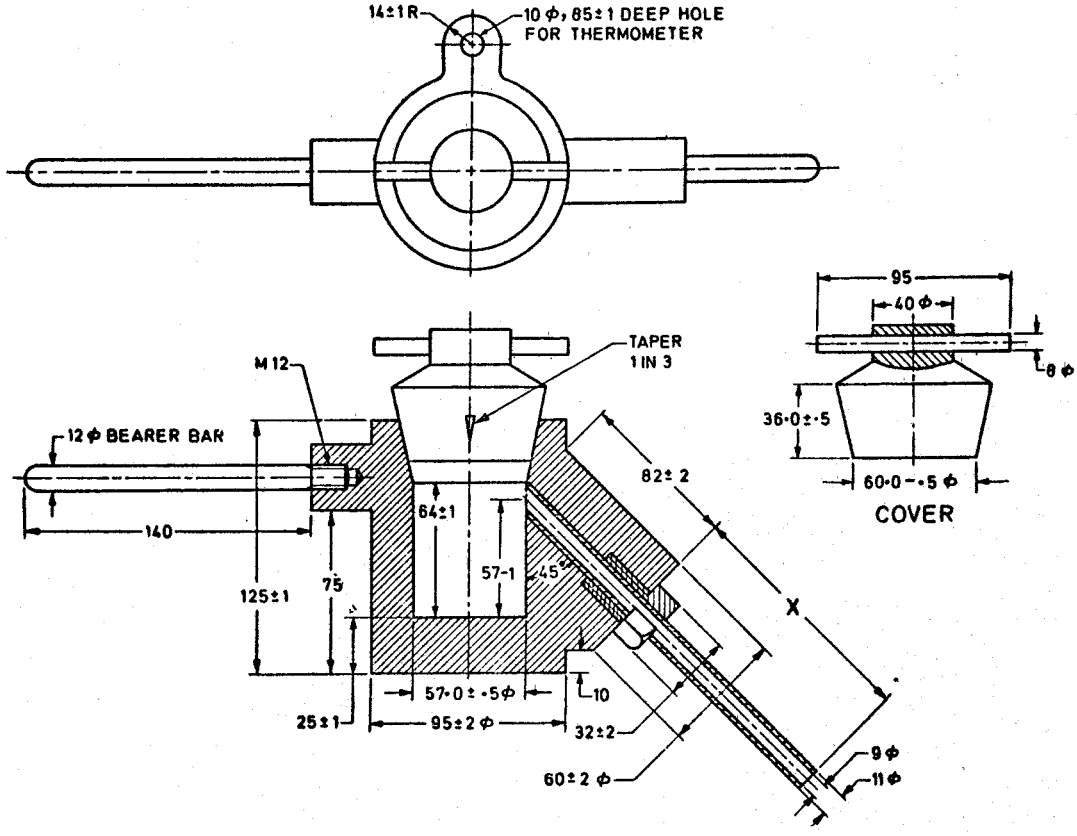
3.3 Temperature Measurement — A thermocouple and millivoltmeter or a nitrogen-filled mercury thermometer, calibrated and capable of indicating up to 550°C. The thermometers should be standardized before use and rechecked at intervals of one month, as follows:

The thermometer, immersed in a metal block at a depth of 10 cm, should be checked by comparing it with a standard thermometer calibrated at an immersion of 10 cm and 60°C temperature of mercury thread. If the differences are greater than ± 3°C a correction factor shall be applied when using such thermometers.

3.4 Receiver — A round-bottomed glass flask of 750 ml capacity with ground-glass joint and with either a long or short neck depending on the method of fitting with the retort as shown in Fig. 2.

3.5 Cooling Bath — A water-bath such that the distance between the flask and the walls of the bath is not less than 2 cm. The water flow is adjusted to maintain a temperature of between 10° and 15°C in the bath.

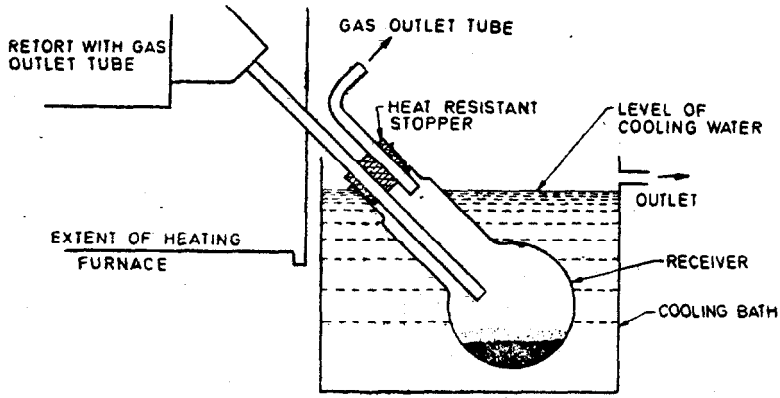
3.6 Distillation Apparatus — A suitable distillation apparatus for the determination of moisture in brown coal and lignite.



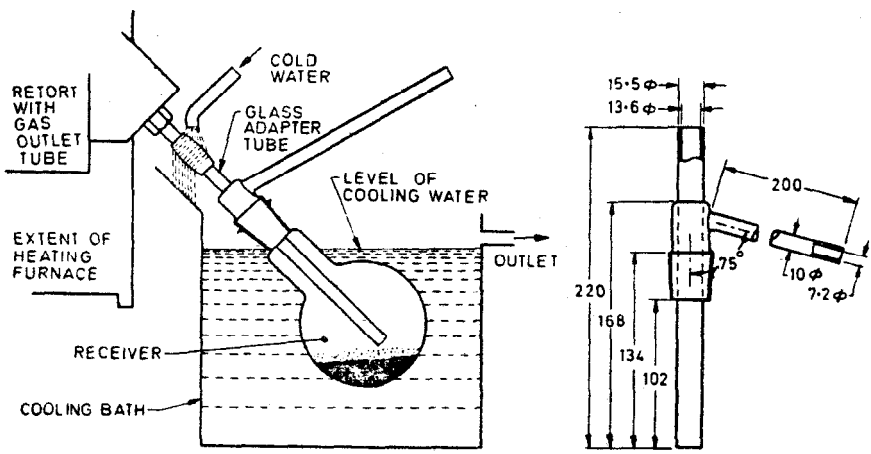
X = Length of exposed section
1) As in Fig. 2A = 195 ± 5 mm
2) As in Fig. 2B = 110 ± 5 mm

All dimensions in millimetres.

FIG. 1 RETORT



2A RECEIVER WITH LONG NECK



2B RECEIVER WITH SHORT NECK

GLASS ADAPTER TUBE

All dimensions in millimetres.

FIG. 2 DISPOSITION OF THE RECEIVER IN THE COOLING BATH

4. REAGENTS

4.1 Graphite — ground, dried and made into a suitable paste with water.

4.2 Toluene — conforming to IS : 1839-1961*.

5. SAMPLE

5.1 Spread the sample on a tray and allow it to attain approximate moisture equilibrium with the atmosphere. Carefully crush the sample so that at least 90 percent passes 1·00-mm IS Sieve (*see* IS : 460-1962†) and not more than 50 percent passes 212-micron IS Sieve. If the moisture content of the crushed sample is still more than 20 percent, further air-drying should be carried out to reduce the moisture content to between 10 and 20 percent. The crushed sample may be stored in a hermetically sealed container. Alternatively, the sample may be kept for not longer than one week in a stoppered container filled to more than 80 percent of its capacity.

NOTE — When samples are kept for longer than one week in containers which are not hermetically sealed or are not entirely filled, the loss of tar yield may be up to 0·5 percent. In certain cases the loss may even be considerably greater.

6. PROCEDURE

6.1 Weigh to the nearest 0·05 g, about 50 g of the sample and transfer it quantitatively to the retort. Lightly smear the conical portion of the cover with the graphite paste, replace the cover and seal by rotating it. Determine the moisture content of the sample at the same time by the volumetric method.

6.2 Weigh the receiver and connect it to the outlet tube of the retort by means of either a heat resistant stopper (Fig. 2A) or a glass adapter tube (Fig. 2B). In the latter case the brass outlet tube should be inserted about 8 mm into the glass adapter tube and sealed by means of a short length of rubber tubing. The joint should be wound with cotton, asbestos, linen, filter paper or similar material and cooled by a stream of water whilst the retort is being heated. Place the retort in the furnace and the receiver in the cooling bath. The receiver shall be immersed in the cooling bath as far as possible, but the rubber stopper or the glass joint should not touch the water. It shall be ensured that the apparatus is gas-tight.

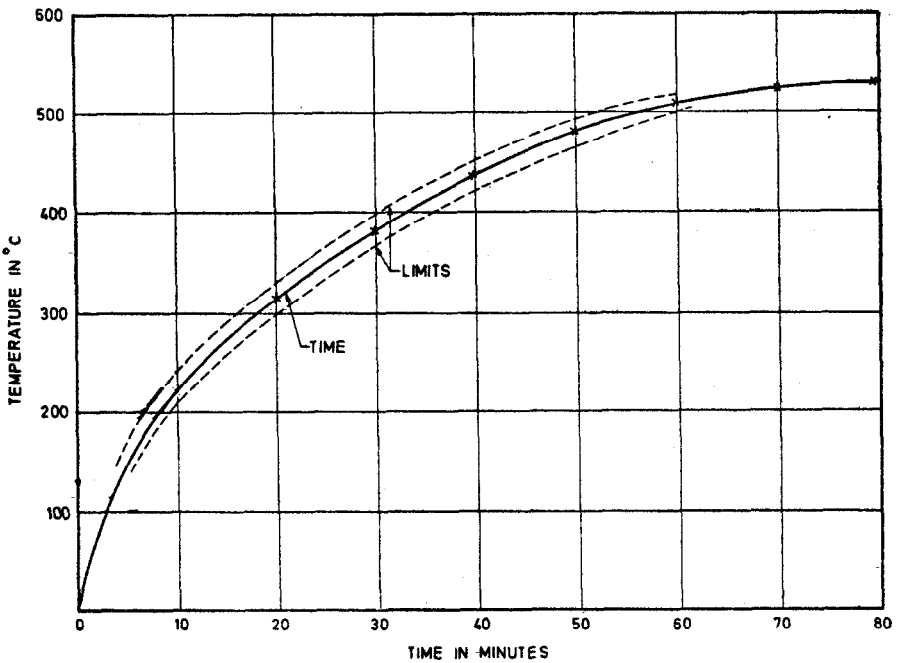
NOTE — It is necessary to preheat certain types of furnace in order to reach 220°C with 10 minutes of inserting the retort.

*Specification for toluene, reagent grade.
†Specification for test sieves (*revised*).

Start the flow of water through the cooling bath and heat the retort according to the following schedule:

Time from Start min	*Temperature* °C
10	220
20	310
30	380
40	440
50	480
60	505
70	520
80	520

The rate of heating may be maintained within the limits shown in Fig. 3.



Total time for low temperature distillation between 20°C and 520°C = 80 minutes
Effective time for low temperature distillation between 310°C and 520°C = 60 minutes

FIG. 3 RISE OF TEMPERATURE DURING DISTILLATION

6.3 Stop heating and remove the retort from the furnace with the receiver still connected; allow to stand for 10 minutes to enable the tar collected in the outlet tube to trickle down into the receiver. Disconnect the receiver from the retort and, if necessary, transfer the remaining tar from the outlet tube into the receiver with a small spatua (only a very small residue of tar will be found in a clean smooth brass tube). Close the outlet tube with a stopper and cool the retort to room temperature. Remove the coke residue carefully and weigh to the nearest 0·05 g in a previously tared weighing bottle.

7. CALCULATION

7.1 The yields on the ' as analysed ' basis are:

a) Coke, percent $= \frac{W_4}{W} \times 100$

b) Tar, percent $= \frac{(W_2 - W_1 - W_3)}{W} \times 100$

c) Water (decomposition), percent $= \frac{W_3}{W} \times 100 - M$

d) Gas (including errors), percent $= 100 - [$ coke + tar + water (decomposition)]

where

W_4 = weight in g of coke residue taken from the retort after the determination, and

W = weight in g of the sample taken for the determination,

W_2 = weight in g of receiver plus tar plus total water,

W_1 = weight in g of empty receiver,

W_3 = weight in g of total water determined by distillation,

M = moisture content of the sample taken for the determination (percent).

The yields on the ' dry ' basis are obtained by multiplying the first three of the above results by $\frac{100}{100 - M}$, the yield of gas is obtained by subtracting M from the fourth result above and then multiplying by $\frac{100}{100 - M}$.

7.2 The result, preferably the mean of duplicate determinations, should be reported to the nearest 0·1 percent. Values for tar, coke, decomposition water and gas should be reported on both the ' as analysed ' basis and on the ' dry ' basis. The tar content may also be calculated on the ' dry ash free ' basis.

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